Transition metal valence in commercial glasses analysed using Xray Absorption Spectroscopy at Balder beamline, MAX IV

THE INDUSTRIAL CHALLENGE

The stone wool manufacturer Paroc (a part of Owens Corning) considers blending in materials into additional waste the production to obtain a more sustainable product. By using waste material that otherwise would go to the landfill also less virgin raw material (volcanic rock) would be used. The waste material contains manganese (Mn) which may potentially affect the iron (Fe) redox equilibria that greatly affects the melt and product properties. Paroc therefor wish to understand and simulate the effect of blending in additional waste material in their product.

WHY USING A LARGE SCALE FACILITY

Low concentrations and extremely complex glassy matrix of stone wool melts makes it impossible to use lab-based methods for analysing the chemical speciation. Labbased method also requires considerable sample preparation, possibly affecting the chemical state. Synchrotron X-ray absorption spectroscopy (XAS) is a powerful method to determine the oxidation state and local structural environment of Mn and Fe. It can be applied to the glassy state of matter and provides the necessary energy to get accurate results. In the future, the BALDER beamline at MAX IV will also provide possibilities for high throughput measurements.

HOW THE WORK WAS DONE

Stone wool is an extremely complex composition matrix and is the reason why six conventional soda-lime-silicate (SLS) glasses containing various known amounts of Mn and Fe were prepared in the laboratory as materials with simpler glassy matrix. In addition, six industrial glass samples were prepared with different blends of waste material. The glass samples were powdered and mixed with polyethylene powder to reduce the concentration of the element to be analysed and bind the powder together so that tablets can be pressed. The experiments were performed at the Balder beamline of MAX IV, Lund, primarily using XANES but some EXAFS spectra were also acquired. The Mn and Fe absorption edge were acquired from 12 glass samples in total. Most were measured in transmission mode, however, for some samples fluorescence mode had to be used due to their low concentration of Mn. In addition, five crystalline Mn/Fe oxide reference materials and the waste material were measured and these were essential for interpreting the XANES results. The absorption edge positions were compared with the reference samples to determine the oxidation states of Fe and Mn in the samples.

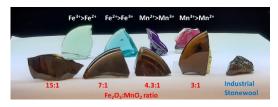


Figure. A photo of selected glassy samples that were measured at Balder (MAX IV).

THE RESULTS AND EXPECTED IMPACT The XANES results show that the redox pair Mn²⁺/Mn³⁺ in oxidized soda-lime-silica melts affects the Fe²⁺/Fe³⁺ redox by increasing the amount of Fe³⁺ in a redox equilibria reaction: $Mn^{3+}+Fe^{2+} \rightleftharpoons Mn^{2+}+Fe^{3+}$. In reduced stone wool melts, there is also an increase in the amount of Fe3+ when Mn is present, however, the increase is not directly reflected by the Mn-concentration and the amount of Mn²⁺ does not necessarily increase simultaneously. The project has led to increased understanding for Paroc on the implications of blending additional waste materials into their production but also an opportunity for Paroc (Owens Corning) to utilize XAS to quantitatively determine the

"A fantastic collaboration giving results from the fascinating facility MAX IV that will be an important step towards a more sustainable stone wool product" /Veronica Sjödin, Paroc AB.



redox equilibria in the glassy state.

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